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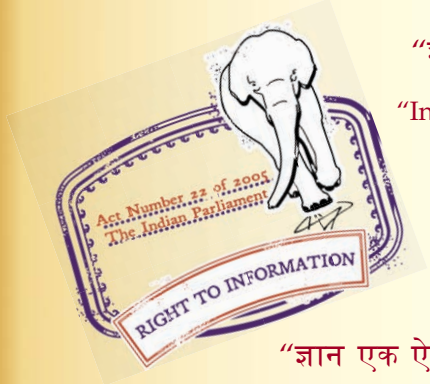

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PART XIX CHLORINE

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METHODS FOR MEASUREMENT OF AIR POLLUTION

PART XIX CHLORINE

0. FOREWORD

0.1 This Indian Standard (Part XIX) was adopted by the Indian Standards Institution on 20 May 1982, after the draft finalized by the Air Quality Sectional Committee had been approved by the Chemical Division Council.

0.2 Chlorine gas is primarily a respiratory irritant. It is so irritating that concentrations above 9 mg/m³ in air are readily detectable by a normal person

0.3 Devices and techniques for determining the concentration of pollutants in the atmosphere are important for the assessment of ' ambient air quality ', establishing hazardous levels in the environment, ameliorative measures and appraisal of contamination from a process or source.

0.4 In the preparation of this standard, considerable assistance has been derived from the Publication No. 42215-01-70 ' Tentative methods of analysis for free chlorine content of the atmosphere (methyl orange method) ' issued by the American Public Health Association.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part XIX) describes the details of methyl orange method for measurement of free chlorine in air.

*Rules for rounding off numerical values (*revised*).

2. METHYL ORANGE METHOD

2.1 Principle — The colour of methyl orange solution ceases to vary with acidity near a *p*H value of 3·0. The dye is quantitatively bleached by free chlorine, and the extent of bleaching may be determined colorimetrically. The optimum concentration range is 0·05-1·0 ppm in ambient air (145 μg to 2 900 μg per m^3 at 25°C and 100 kPa).

NOTE — It is essential to maintain proper *p*H

2.2 Range and Sensitivity — The procedure given is designed to cover the range of 5-100 μg of free chlorine per 100 ml of sampling solution. For a 30 litre air sample, this corresponds to approximately 0·05-1·0 ppm in air which is the optimum range. Increasing the volume of air samples extends the range at the lower end, but only within limits, since 50 litre of chlorine free air produces the same effect as about 0·01 ppm of chlorine.

2.3 Limitations and Interferences — Free bromine, which gives the same reaction, interferes in a positive direction. Manganese (III, IV) in concentrations of 0·1 ppm or above also interferes positively. Negative interference from sulphur dioxide is significant both in solution as well as gaseous state. Nitrites impart an off-colour orange to the methyl orange reagent. Nitrogen dioxide interferes positively, reacting as 20 percent chlorine. Sulphur dioxide interferes negatively, decreasing the chlorine by an amount equal to one third the sulphur dioxide concentration.

2.4 Precision and Accuracy — Error due to measurement by this procedure is known to be less than $\pm$ 5 percent of the amount present.

2.5 Apparatus

2.5.1 *Spectrophotometer* — suitable for measurement at 505 nm, preferably accommodating 5-cm cells.

2.5.2 *Sampling Train* — according to IS : 5182 (Part V)-1975* using a large impinger with fritted disc [*see* Fig. 2 of IS : 5182 (Part V)-1975*] of porosity B (70 to 100 μm maximum pore diameter).

2.6 Reagents — Pure chemicals of analytical grade, unless otherwise specified, and distilled water (*see* IS : 1070-1977†) shall be employed in the test. These shall not contain any impurities that affect the results of analysis.

2.6.1 *Methyl Orange Stock Solution 0·05 Percent* — Dissolve 0·500 g of methyl orange in water and dilute to one litre. This solution is stable indefinitely if freshly boiled and cooled distilled water is used.

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*Methods for measurement of air pollution : Part V Sampling of gaseous pollutants.
†Specification for water for general laboratory use (*second revision*).

2.6.2 *Methyl Orange Reagent 0·005 Percent* — Dilute 100 ml of stock solution to one litre with water. Prepare fresh for use.

2.6.3 *Sampling Solution* — Dilute 6 ml of 0·005 percent methyl orange reagent to 100 ml with water and add three drops (0·15-0·20 ml) of 5 N hydrochloric acid. One drop of butanol may be added to induce foaming and increase collection efficiency. *p*H of the solution may be checked using a *p*H meter.

2.6.4 *Acidified Water* — Add three drops (0·15-0·20 ml) of 5 N hydrochloric acid to 100 ml of water.

2.6.5 *Potassium Dichromate Solution — 0·100 0 N* — Dissolve 4·904 g anhydrous potassium dichromate primary standard grade in water and dilute to one litre.

2.6.6 *Starch Indicator Solution* — Prepare a thin paste of 1 g of soluble starch in a few millilitres of water. Bring 200 ml of water to boil, remove from heat, and stir in the starch paste. Prepare fresh before use.

2.6.7 *Potassium Iodide* — reagent grade.

2.6.8 *Sodium Thiosulphate Solution 0·1N* — Dissolve 25 g of $Na_2S_2O_3.5H_2O$ in freshly boiled and cooled water and dilute to one litre. Add 5 ml chloroform as preservative and allow to age for two weeks before standardizing. Take 80 ml water, 1 ml sulphuric acid and 1 g of potassium iodide in a titration flask and pipette into this, with constant stirring, 10 ml of 0·100 0 N potassium dichromate solution. Allow to stand in the dark for 6 minutes. Titrate with 0·1 N thiosulphate solution. Upon approaching the end-point (brown colour changing to yellowish green), add 1 ml of starch indicator solution and continue titrating to the end-point (blue to light green).

$$\text{Normality of } Na_2S_2O_3 = \frac{1{\cdot}000\ 0}{V}$$

where

V = volume in ml of sodium thiosulphate solution used.

2.6.9 *Sodium Thiosulphate Solution 0·01 N* — Dilute 100 ml of the aged and standardized 0·1 N sodium thiosulphate solution to 1 litre with freshly boiled and cooled distilled water. Add 5 ml chloroform as preservative and store in a glass-stoppered bottle. Standardize frequently with 0·010 0 N potassium dichromate.

2.6.10 *Chlorine Solution, Approximately 10 ppm* — Prepare by serial dilution of household bleach (approximately 50 000 ppm), or by dilution of strong chlorine water made by the bubbling chlorine gas through cold

distilled water. The diluted solution should contain approximately 10 ppm of free (available) chlorine. Prepare one litre.

2.7 Procedure — Assemble the sampling train. Add 100 ml of sampling solution to the impinger and draw a measured volume of air at a rate of 1-2 l/min for a period of time appropriate to the estimated chlorine concentration. Transfer the solution to a 100-ml volumetric flask and make to volume, if necessary, with acidified water. Measure absorbance at 505 nm in 5-cm cells against water as reference.

2.7.1 The volume of sampling solution, the concentration of methyl orange in the sampling solution, the amount of air sampled, the size of impinger and the length of photometer cell may be varied to suit the needs of the situation as long as proper attention is paid to the corresponding changes necessary in the calibration procedure.

2.8 Calibration

2.8.1 Prepare a series of six 100-ml volumetric flasks containing 6 ml of 0·005 percent methyl orange reagent, 75 ml water, and 3 drops (0·15-0·20 ml) of 5·0 N hydrochloric acid. Carefully and accurately pipette 0, 0·5, 1·0, 5·0 and 9·0 ml of chlorine solution (approximately 10 ppm) into the respective flasks; holding the pipette tip beneath the surface. Quickly mix and make to volume with acidified water.

2.8.2 Immediately standardize the 10 ppm chlorine solution by adding 400 ml of chlorine solution to a flask containing 1 g potassium iodide and 5 ml glacial acetic acid, and swirling to mix. Titrate with 0·01 N sodium thiosulphate until the iodine colour becomes a faint yellow. Add 1 ml of starch indicator solution and continue the titration to the end-point (blue to colourless). One millilitre of 0·010 0 N sodium thiosulphate is equivalent to 0·354 6 mg of free chlorine. Calculate the amounts of free chlorine added to each flask in **2.8.1**.

2.8.3 Transfer the standards prepared in **2.8.1** to absorption cells and measure absorbance against micrograms of chlorine to draw the standard curve.

2.9 Calculations — Calculate the chlorine concentration as follows:

$$\text{Chlorine, } \mu g/m^3 = \frac{M}{V}$$

where

M = amount of chlorine found, μg; and

V = volume of air sampled, m^3.

For different temperatures and atmospheric pressures, proper correction for air volume should be made.

2.10 Effect on Storage — The colour of sampled solutions is stable for at least 24 h if protected from direct sunlight, although the presence of certain interferences (Fe III) may cause slow colour change.

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